I. Instrument Name: LEO Scanning Electron Microscope/Oxford Energy Dispersive X-ray System (SEM/EDS)

II. Suggested Uses: High resolution and magnification imaging with enhanced depth of field for trace evidence. Non-destructive elemental analysis of gunshot residue particles, paint, metal, powders, and other trace particulate material.

III. Operating Procedures

A. Start-up procedures (Consult manuals and Help Menu for Leo 1450 Variable Pressure SEM and Oxford Energy Dispersive X-ray System)

1. SEM

a. Engage the SEM Green "ON" button on the front of the plinth (DO NOT TOUCH the "OFF" button which is red in color).

b. After the visual screen for the SEM (TV monitor 2) appears, enter your name and password as requested (You may have to use CRTL-ALT function and 2 in order to access screen 2).

c. Open the LEO USER INTERFACE 2 icon. Enter your name and password. SEM screen will appear. All commands for the SEM are presented/accessed through this screen. The mouse is used to activate or deactivate each command.

d. Changing Samples.

(1) Be sure that the filament and extra high tension (EHT) are "OFF." (Go to Status Toolbar, EHT or Filament options.)

(2) Vent Chamber

(a) Click on the Vacuum option on the Status Toolbar

(b) Click on Vent. Select "YES" for venting.

(c) Unlatch both chamber door latches and adjust nitrogen pressure to 3-4 psi.

(3) Turn off nitrogen supply

(4) Pull out the chamber door with the attached stage (Be careful to clear cables near the door).'

(5) Remove the stage holder and place it on a clean surface (use gloves or touch the holder as little as possible).

(6) Place samples in the holder (noting position of each sample)

(7) Tighten the screws for each mount position

(8) Place the holder back on the stage exactly as you removed it, ensuring that the flat edge of the mount is against the flat area of the stage.

(9) Roll the stage back into the chamber and securely latch the

chamber door latches

(10) Pump the chamber back to Vacuum Ready Condition by using vacuum selection on the Status Toolbar.

e. Activate the EHT and Filament using the Status Toolbar. The last saved working conditions will be automatically brought up for the filament.

f. Check the emission mode to determine if the filament is properly aligned and saturated.

(1) Adjust emission image and filament by going to User Toolbar, Beam, Gun Alignment

(2) Use the electronic positioning adjustments to center the fully saturated emission image.

(3) Then adjust saturation of the filament

(a) For the best image, set saturation slightly below or at the second crossover

(b) For lower quality image and typical probe work, a saturation point between the first and second crossover is usually recommended

(c) The lower the saturation, the longer the filament life.

g. Adjust working parameter of the instrument.

(1) Many can be accessed through the User Toolbar, Tools, "Go to Control Panel"

2. EDS

a. Power On

(1) Via the HP computer located in lower right area of the EDS table.

(2) Depressing the power button will bring up the HP EDS monitor (our Monitor 1)

(a) The power button is the larger one on the far right of the computer.

(3) Log on to EDS system

(a) CTRL-ALT 1, then CTRL-ALT DEL to log in

(b) Enter your name and password

(c) Choose between the two platforms

i) ISIS (used for automated Gunshot Residue Analysis)

ii) INCA

B. Collection and Storage of Data

1. SEM

a. Monitor the parameters of the instrument to determine if a different accelerating voltage, current setting, signal collector, or display variable would enhance the image on the display monitor.

b. Perform sizing of particles if needed.
c. Save image (if needed)
(1) Label pertinent information on the monitor using Annotation drop down menu
(2) Save both the image and annotation to the hard drive.
d. Print using the high resolution Codonics printer (if needed)

2. EDS
a. Set collection time and acquisition rate using the Edit function in Isis.
(1) Use Acquisition Rate 6 for most analysis except Gunshot Residue Analysis (Rate 4) and mapping (Rate 1-2).
(2) Analysis time will vary with 200 seconds being an average time.
b. Adjust dead time
(1) Click on X-ray start button and collect enough counts to obtain about 30% dead time.
(2) Adjust dead time using SEM spot size.
(3) Stop x-ray counting as soon as dead time adjustments are made.
c. Collect Spectra
(1) Click on start and collect an x-ray spectra for the preset time.
(2) During and after collection you may expand different regions of the spectra and identify spectral peaks either automatically or manually.
(3) Once spectral peaks are identified and confirmed, save to the hard drive and/or print on the Epson color printer.

C. Calibration of the SEM & EDS
1. See Calibrations manual for procedures.

D. Shut-down Procedures
1. SEM
a. The SEM vacuum always remains on. NEVER LEAVE THE SYSTEM FOR EXTENDED PERIODS EXCEPT AT HIGH VACUUM.
b. Close the SEM operating window and answer the dialog questions.
c. Close down the SEM user interface.
d. Shut down the computer using the Start icon in the lower left corner of the monitor.
e. Once closed, you may press yellow STANDBY button on the front of the column (or plinth).
(1) This will keep the electronics on but in a standby mode.
(2) If you press the red OFF button, you will remove all the voltage to the instrument and will lose stage settings.

Therefore, never press the red OFF button except for major resetting of the instrument or for servicing of the syste.

(3) CAUTION: If you are going to work on the electronics of the instrument, be sure to also press the red OFF button so that the 220V current to the instrument is removed. Failure to do this could be very dangerous to one's health.

2. EDS
   a. If the x-ray detector is still counting, click on the STOP function to terminate counting by the detector. Save spectra if needed.
   b. Close out all windows in ISIS or INCA.
   c. Shut down the computer.

IV. Safety Concerns
   A. EDS x-rays
   B. Liquid nitrogen used to cool the detector of the EDS can be dangerous to the skin and eyes if exposed for prolonged periods.
      1. It is recommended to use safety gloves and eye protection when filling the dewar of the EDS system manually.
      2. An automatic Liquid Nitrogen Fill System is in place which should prevent having to manually fill the liquid nitrogen dewar.